# UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION II

**DATE: January 11, 2008** 

JAN 15 PM 12: 08

SUBJECT: Tissue Chemical Data Assessment - Sampling and Testing of Material Proposed for Dredging from the

San Juan Harbor, Puerto Rico.

FROM: Carol L. Lynes, Environmental Scientist

Monitoring and Assessment Branch

To: Mark Reiss, Environmental Scientist Dredging Sediments and Ocean Team

As per your request, I have completed my assessment of the tissue chemical data summaries produced for the San Juan Harbor Dredging Project. Specific review criteria and comments are included in the attached chemical data assessment report.

The assessment of the chemical data associated with this project concludes that the data are "acceptable" and "acceptable with condition". Details of the data acceptability determinations are provided in the attached report narrative and tables.

If you have any questions, please feel free to contact me at 732-321-6760 or <a href="mailto:lynes.carol@epa.gov">lynes.carol@epa.gov</a>.

Attachment

Cc. J. Ferretti, DESA-LAB

2008 JAN 15 PM 1:07 D.S. EPA-REGION 2 BLEANWATER REGULATORY BR.

## SUMMARY OF FINDINGS AND TECHNICAL RECOMMENDATIONS - CHEMICAL TESTING

**Project(s):** Dredged Material Testing for Proposed Ocean Disposal – San Juan Harbor, Puerto Rico **Chemical Testing Laboratories:** Battelle Laboratories

Chemical testing was conducted on tissue matrices for PAH Compounds, Pesticides, PCB Congeners, and Metals. Tissue summary data, including quality control results, were reviewed for compliance with the established testing requirements as specified below.

#### Tissue samples

Procedural/Method Blank - One per batch of 1-20 samples

Matrix spike (in Triplicate) - Fortified 3 to 5x the RL identified in the manual; one set per batch of 1-20 samples

LCS/LFB - Fortified to 3 to 5x the RL identified in the manual; one per batch of 1-20 samples SRM - Not required

Surrogate Spike - per sample (Organics Only)

Initial Calibration Check Samples with calculated results of the recoveries.

Continuing Calibration Check Samples with calculated results of the recoveries.

The assessment and conclusions on data acceptability are outlined in the following tables. The acceptability of the data for each chemical group and matrix is categorized as "acceptable", "acceptable with condition", "unacceptable", and/or "not determined". It is our opinion that the data with an "acceptable" or "acceptable with condition" may be used; however, if indicated, please note the particular condition. The category "not determined" is used if insufficient data were provided.

## **Reporting Comments:**

For future submittals, Anamar must include copies of the analytical results data sheets for all samples, including the quality control samples.

### **Laboratory Analysis Comments:**

Pesticides/PCBs – The initial calibration check samples for all analytical batches did not meet the established acceptance criterion.

07-0043 Congener 206 exceeded the criterion.

07-0048 Congeners 206 and 209 exceeded the criterion.

07-0049 Congeners 206 and 209exceeded the criterion.

PAHs – The matrix spike, matrix spike duplicate, and matrix spike triplicate samples were over-fortified in all analytical batches. Over-fortified spike samples will not be accepted.

Metals (Ag. As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)

Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zii)					
QC Measurement	Frequency	Calculation	Acceptance Criteria	<u>Tissue</u>	
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.	
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery And %RSD	70 -130% (Recovery) 30% (RSD)	<ul> <li>M. nasuta: All results are within the acceptance criteria.</li> <li>N. virens: Zn was recovered below 70% in the MS and the MST. The low recovery resulted in an RSD exceeding 30%. All remaining results are within the acceptance criteria.</li> </ul>	
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	% Recovery *Evaluated for analytes > 3x RL	70 - 130%	M. nasuta: Not required for tissue analysis.  N. virens: Not required for tissue analysis.	
LCS/LFB	1 per 20 samples	% Recovery	70 -130%	M. nasuta: All results are within the acceptance criterion.  N. virens All results are within the acceptance criterion.	
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	90 - 110%	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	
Continuing Calibration Checks using Calibration Standards	Minimum – check calibration at middle and end of each batch or 1 per 10 analyses, which ever is greater	% Recovery	90 - 110% from initial calibration for each analyte	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	
Data Acceptability	M. nasuta: Acceptable.  N. virens: Acceptable with condition that the results for zinc are biased low.				

Organics (Pesticides, PCBs) Analytical Batch: 07-0043

QC Measurement	Frequency	Calculation	QC Control Acceptance Criteria	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	N. virens: All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	N. virens: All results are within the acceptance criteria.
Standard Reference Material (SRM)	1 per 20 samples	%Recovery	50 - 150%	N. virens: Not required for tissue analysis.
(Sediment/Water Only)		*Evaluated for analytes >3x the RL		
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	N. virens: All results are within the acceptance criterion.
Surrogate Standards	Each sample		30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	N. virens: All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve			N. virens: Congener 206 exceeds the criteria. The bias for this congener is unknown.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	initial calibration for each analyte	N. virens: All results are within the acceptance criterion.
Data Acceptability	N. virens: Acceptable	with the condition	that the result for cond	gener 206 is to be considered estimated. All remaining congeners are acceptable.

Organics (PAHs) Analytical Batch: 07-0043

Calculation   Acceptance   Criteria   Criteria	Organic	CS (PARS)	Arialytical Dat		T-1
Laboratory Method Blank   1 per 20 samples   NA   NA   NA   NA   NA   NA   NA   N	QC Measurement	Frequency	Calculation	Acceptance Criteria	Tissue
Laboratory Method Blank  Matrix Spikes (Triplicate)  1 set per 20 samples  Standard Reference Material (SRM) (Sediment/Water Only)  LCS/LFB  1 per 20 samples  1 per 20 samples  NA  Standard's  Surrogate Standard's  Initial Calibration Check Standard's  Continuing Continuing Calibration Standards  Standards  NA  Stecovery and Shade Recovery (Recovery) Shade Recovery Shade Recovery Shade Recovery (Recovery) Shade Recovery Shade Reference Sha	Holding Time	NA	I NA	•	
Criplicate   Samples   Solution   Samples   So	SIMPLE STATE OF THE STATE OF TH	1 per 20 samples	NA	No analyte should be detected at > RL	
Standard Reference Material (SRM) (Sediment/Water Only)  LCS/LFB  1 per 20 samples  Recovery (%R)  Surrogate Standards  Surrogate Standards  Initial Calibration Check Standards Continuing Calibration Checks using Calibration Standards Standards  Standards  A per 20 samples  Recovery (%R)  Recovery (%R)  So -150%  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	***************************************	/6)	and	(Recovery)	acceptable, even though all of the results are within the acceptance criterion. Over-fortified spike samples will not be accepted.
CS/LFB		1 per 20 samples		50 - 150%	N. virens: Not required for tissue analysis.
LCS/LFB 1 per 20 samples % Recovery (%R)  Surrogate Standards Each sample Each sample % Recovery % all applicable surrogate standards must be within the acceptance range to be considered acceptable % Recovery following calibration curve % Recovery % Rec			analytes >3x the		All and the are within the eccentance criterion
Surrogate Standards  Each sample  We Recovery  Surrogate Standards  Surrogate Surrogate Standards  Surrogate	LCS/LFB	1 per 20 samples		50 -150%	
Initial Calibration Check Standards  Immediately following calibration curve  Continuing Calibration Checks using Calibration Standards  Immediately following calibration curve  % Recovery some calibration for each batch or 1 per 10 analyses, which ever is greater.  % Recovery some calibration shows a standards  % Recovery some calibration for each initial calibration for each analyte  % Recovery some calibration for each initial calibration for each analyte	Surrogate Standards	Each sample		* all applicable surrogate standards must be within the acceptance range to be considered acceptable	
Continuing Minimum - middle Calibration Checks using Calibration batch or 1 per 10 Standards analyses, which ever is greater.  Minimum - middle % Recovery initial calibration for each analyte  80 – 120% from initial calibration for each analyte		following	% Recovery		
The state of the s	Calibration Checks using Calibration	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.		initial calibration for	N. virens: All results are within the acceptance criterion.
Data Acceptability  N. virens: Acceptable.	Data Acceptability	N. virens: Acceptab	ele.		

Organics (Pesticides, PCBs) Analytical Batch: 07-0048

Organics (resticides, PCBS) Analytical Batch: 07-0048				
QC Measurement	Frequency	Calculation	QC Control Acceptance Criteria	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	M. nasuta: All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	M. nasuta: All results are within the acceptance criteria.
Standard Reference Material (SRM)	1 per 20 samples	%Recovery	50 - 150%	M. nasuta: Not required for tissue analysis.
(Sediment/Water Only)		*Evaluated for analytes >3x the RL		
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	M. nasuta: All results are within the acceptance criterion.
Surrogate Standards	Each sample	,	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	M. nasuta: All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	M. nasuta: Congeners 206 and 209 exceed the criteria. The bias for these congeners is unknown.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	initial calibration for each analyte	M. nasuta: All results are within the acceptance criterion.
Data Acceptability	M. nasuta: Acceptable	with the condition	that the results for co	ngeners 206 and 209 are to be considered estimates. All remaining congeners are acceptable.

Organics (PAHs) Analytical Batch: 07-0048

Organi	CS (PAHS)	Analytical bat		
QC Measurement	Frequency		QC Control Acceptance Criteria	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	M. nasuta: All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	M. nasuta: The spiked samples were over-fortified. The MS, MSD, MST samples are not acceptable, even though all of the results are within the acceptance criterion. Over-fortified spike samples will not be accepted.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	*Evaluated for analytes >3x the RL	50 - 150%	M. nasuta: Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	M. nasuta: All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	M. nasuta: All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	M. nasuta: All results are within the acceptance criterion.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	M. nasuta: All results are within the acceptance criterion.
Data Acceptability	M. nasuta: Acceptal	ble.		

Organics (Pesticides, PCBs) Analytical Batch: 07-0049

QC Measurement	Frequency	Calculation	QC Control Acceptance Criteria	<u>Tissue</u>	
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.	
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	M. nasuta: All results are within the acceptance criteria.  N. virens: All results are within the acceptance criteria.	
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	M. nasuta: Not required for tissue analysis.  N. virens: Not required for tissue analysis.	
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	
Surrogate Standards	Each sample		30-150%  * all applicable surrogate standards must be within the acceptance range to be considered acceptable	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.	
Initial Calibration Check Standards	Immediately following calibration curve		80 – 120%	M. nasuta/N. virens: Congeners 206 and 209 exceed the criteria. The bias for these congeners is unknown.	
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	initial calibration for each analyte	M. nasuta/N. virens: All results are within the acceptance criterion.	
	M. pasuta: Acceptable with the condition that the results for congeners 200 and 200 and the				

Organics (PAHs) Analytical Batch: 07-0049

Organii	S (PARS) Analyti	cui Batom C.	00.0	Ticquo
QC Measurement	Frequency	<u>Calculation</u>	Acceptance Criteria	<u>Tissue</u>
Holding Time	NA	NA	Method Specific	All samples were prepared and analyzed within the established holding times.
Laboratory Method Blank	1 per 20 samples	NA	No analyte should be detected at > RL	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.
Matrix Spikes (Triplicate)	1 set per 20 samples	% Recovery and %RSD	50 - 150% (Recovery) 50% (RSD)	M. nasuta/N. virens: The spiked samples were over-fortified. The MS, MSD, MST samples are not acceptable, even though all of the results are within the acceptance criterion. Over-fortified spike samples will not be accepted.
Standard Reference Material (SRM) (Sediment/Water Only)	1 per 20 samples	%Recovery *Evaluated for analytes >3x the RL	50 - 150%	M. nasuta: Not required for tissue analysis.  N. virens: Not required for tissue analysis.
LCS/LFB	1 per 20 samples	% Recovery (%R)	50 -150%	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.
Surrogate Standards	Each sample	% Recovery	30-150% * all applicable surrogate standards must be within the acceptance range to be considered acceptable	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.
Initial Calibration Check Standards	Immediately following calibration curve	% Recovery	80 – 120%	M. nasuta: All results are within the acceptance criterion.  N. virens; All results are within the acceptance criterion.
Continuing Calibration Checks using Calibration Standards	Minimum - middle and end of each batch or 1 per 10 analyses, which ever is greater.	% Recovery	80 – 120% from initial calibration for each analyte	M. nasuta: All results are within the acceptance criterion.  N. virens: All results are within the acceptance criterion.
Data Acceptability	M. nasuta: Acceptate N. virens: Acceptable		*	